

Europäisches Patentamt

European Patent Office

Office européen des brevets



EP 0 668 152 B1 (11)

(12)

EUROPEAN PATENT SPECIFICATION

(45) Date of publication and mention of the grant of the patent: 23.12.1998 Bulletin 1998/52

(51) Int. Cl.6: B31F 1/07, A47K 10/16, D21F 11/00

(21) Application number: 95400317.4

(22) Date of filing: 15.02.1995

(54) Method for making soft high bulk tissue and tissue products obtained

Verfahren zum Herstellen von hoch gebauschtem Papier und Papierprodukte daraus hergestellt Procédé pour faire du papier bouffant doux p. ex.: mouchoirs en papier et produits en papier ainsi obtenus

- (84) Designated Contracting States: BE DE ES FR GB IT NL SE
- (30) Priority: 18.02.1994 US 195762
- (43) Date of publication of application: 23.08.1995 Bulletin 1995/34
- (73) Proprietor: KIMBERLY-CLARK WORLDWIDE, INC. Neenah, Wisconsin 54956 (US)
- (72) Inventors:
 - · Kamps, Richard Joseph Wrightstown, Wisconsin 54180 (US)
 - · Behnke, Janica Sue Appleton, Wisconsin 54915 (US)

- · Chen, Fung-jou Appleton, Wisconsin 54915 (US)
- · Radtke, Darnell Clarence Shiocton, Wisconsin 54170 (US)
- (74) Representative: Sauvage, Renée **Cabinet Sauvage** 100 bis, avenue de Saint-Mandé 75012 Paris (FR)
- (56) References cited:

EP-A- 0 117 351	EP-A- 0 303 528
EP-A- 0 426 288	EP-A- 0 475 671
EP-A- 0 613 979	WO-A-85/03029
WO-A-94/24366	DE-B- 1 235 126
GB-A- 2 166 690	US-A- 4 339 088
US-A- 4 671 983	

Note: Within nine months from the publication of the mention of the grant of the European patent, any person may give notice to the European Patent Offic of opposition to the European patent granted. Notice of opposition shall be filed in a written reasoned statement. It shall not be deemed to have been filed until the opposition fee has been paid. (Art. 99(1) European Patent Convention).

Descripti n

In the manufacture of soft tissue products such as facial, bath and towel tissue, an aqueous suspension of paper-making fibers is deposited onto a forming fabric from a headbox. The newly-formed web is thereaft r dewatered, dried and creped to form a soft tissue sheet. The trend in premium tissue manufacture has been to provide softer, bulkier, less stiff sheets by layering, throughdrying and basis weight reductions. Layering, which requires a headbox equipped with headbox dividers, enables the tissue manufacturer to engineer the tissue by placing softer feeling fibers in the outer layers while placing the stronger fibers, which generally do not feel as soft, in the middle of the tissue sheet. Throughdrying enables the manufacturer to produce a bulky sheet by drying the sheet with air in a noncompressive state. Reducing the basis weight of the sheet reduces its stiffness and, when used in conjunction with throughdrying, a single-ply tissue sheet of adequate caliper and performance for a premium product can be attained.

However, producing a premium tissue product of adequate softness, bulk and strength on conventional (wetpressed) tissue machines is not easily accomplished. For example, layering requires the purchase of a layered headbox, which is expensive. Higher bulk can be achieved by embossing, but embossing normally requires a relatively stiff
sheet in order for the sheet to retain the embossing pattern. Increasing sheet stiffness negatively impacts softness.

Conventional embossing, such as that carried out in WO 85/03029, also substantially reduces the strength of the sheet
and may lower the strength below acceptable levels in an effort to attain suitable bulk. US-A-4 339 088 is directed to
another method of embossing, in which products in convolutely wound rolls are embossed with co-mating rolls. Reducing the basis weight of the sheet will decrease its stiffness, but may require that two or more of such low basis weight
sheets be plied together to retain the desired caliper and performance. In terms of manufacturing economy, multiple-ply
products are more expensive to produce than single-ply products, but single-ply products generally lack sufficient softness and bulk, especially when manufactured on conventional machines.

Accordingly there is a need for a simple means of enabling conventional tissue machines to produce premium quality tissue sheets having adequate softness, bulk and strength without the expense of purchasing a layered headbox or a throughdryer, or manufacturing multiple plies.

Definitions

35

55

As used herein, Strength is the geometric mean tensile (GMT) strength, which is the square root of the product of the machine direction (MD) tensile strength and the cross-machine direction (CD) tensile strength of the tissue sheet. The MD tensile strength, MD stretch, CD tensile strength, and CD stretch are determined in accordance with TAPPI test method T 494 om-88 using flat gripping surfaces (4.1.1, Note 3), a jaw separation of 50.8 millimeters (or 2.0 inches), a crosshead speed of 254 millimeters (or 10 inches) per minute. The units of Strength are grams per 76.2 millimeters (or 3 inches) of sample width, but for convenience are herein reported simply as "grams".

The Bulk of the products of this invention is calculated as the quotient of the Caliper (hereinafter defined), expressed in microns, divided by the basis weight, expressed in grams per square meter. The resulting Bulk is expressed as cubic centimeters per gram.

The Caliper, as used herein, is the thickness of a single sheet, but measured as the thickness of a stack of ten sheets and dividing the ten sheet thickness by ten, where each sheet within the stack is placed with the same side up. It is measured in accordance with TAPPI test methods T402 "Standard Conditioning and Testing Atmosphere for Paper, Board, Pulp Handsheets and Related Products" and T411 om-89 "Thickness (Caliper) of Paper, Paperboard, and Combined Board" with Note 3 for stacked sheets. The micrometer used for carrying out T411 om-89 is a Bulk Micrometer (TMI Model 49-72-00, Amityville, New York, USA) having an anvil pressure of 3.39 kilopascals (220 grams per square inch) and an anvil diameter of 103.2 millimeters (4-1/16 inches). After the Caliper is measured, the same ten sheets in the stack are used to determine the average basis weight of the sheets.

As used herein, Specific Elastic Modulus (SEM) is determined by measuring the slope of a particular portion of the machine-direction stress/strain curve for the tissue in question. The SEM is calculated as the slope of the machine direction stress/strain curve (expressed In kilograms per 76.2 millimeters of sample width) measured between a stress of 100 and 200 grams, divided by the product of 0.0762 times the basis weight (expressed in grams per square meter). The SEM is expressed in kilometers and is an objective measure of tissue softness.

The "embossing level" is the distance a male element of a male embossing roll penetrates the corresponding female void of a female embossing roll.

The "accommodation" is the distance between the sidewalls of the male elements and the sidewall of the female voids at zero engagement.

It has now been discovered that a strong, soft and bulky tissue sheet of premium quality can be produced from basesheets made with conventional tissuemaking assets, although the method of this invention can also be used to improve premium quality basesheets as well. (As used herein, a tissue "basesheet" is a tissue sheet as produced on a tissue machine and wound up, prior to any post treatment such as the embossing method of this invention. The tissue

basesheet can be layered or blended, creped or uncreped. A tissue "sheet" is a single-ply sheet of tissue, which can be a tissue basesheet or a post-treated tissue basesheet. A tissue product is a final product consisting of one or more tissue sheets.) A premium quality tissue sheet has a Strength of 500 grams or greater, a Bulk of 6 cubic centim ters per gram or greater, and a softness, as measured by the Specific Elastic Modulus of 4 kilometers or less. The invention utilizes a debonding method in which fine-scale, discrete, interm shing embossing elements of two gendered (male and female) embossing rolls inelastically strain the tissue sheet, thereby rupturing the weak bonds and opening up the structure both internally and externally. When the method of this invention inelastically strains the sheet externally, the sheet has increased surface fuzziness, which can improve softness. When the method of this invention inelastically strains the sheet internally, the sheet is more limp (less stiff) with a lower Specific Elastic Modulus (increased softness) and significantly greater Bulk. In most cases, the Strength of the sheet is substantially unaffected. Depending on the properties of the sheet to which the method of this invention is applied, the resulting product will have different characteristics, but will always be improved in terms of softness and Bulk, preferably without significant loss of Strength.

New and different tissue sheets and multi-ply tissue products are produced when the method of this invention is applied to wet-pressed or throughdried tissue sheets, including layered or nonlayered (blended) tissue sheets. When the method of this invention is applied to certain blended tissue sheets (wet-pressed or throughdried), softness properties which closely approach the softness characteristics of layered tissue sheets can be obtained by increasing the number of unbonded fiber ends protruding from the surface of the tissue sheet. When the method of this invention is applied to wet-pressed tissue sheets (either layered or blended), the Bulk and softness are improved to the point of being comparable to that of throughdried sheets. For purposes herein, an increase in softness is objectively represented by a decrease in the Specific Elastic Modulus (SEM), which is a measure of stiffness. In all cases, the Strength of the sheet or product is maintained at a useful level of about 500 grams or greater.

Hence in one aspect the invention resides in a method of embossing a tissue sheet comprising passing a tissue sheet through a nip formed between male and female embossing rolls having about 15 or more discrete, intermeshing, unmatched embossing elements per square centimeter (100 per square inch) of surface which deflect the sheet perpendicular to its plane. Rolls of this type are already known from WO 94/24366 which is a prior art document according to Article 54(3) EPC. This document refers to a micro-straining process that is intended to increase the internal bulk of a tissue sheet.

The inventive feature of the method according to this invention is that the embossing elements cause, when they are engaged, shearing of the tissue to create a permanent deformation without perforation of said tissue, so that the percent increase in Bulk divided by the percent decrease in Strength is about 1 or greater, more specifically from about 1 to about 4, and still more specifically from about 2 to about 3.

The method of this invention unexpectedly can substantially increase the number of unbonded fiber ends protruding from the surface of the tissue sheet for certain blended basesheets which are debondable, meaning that a significant percentage of the papermaking bonds in the sheet are weaker than the fiber strength. When strained, the fiber-to-fiber bonds in the basesheet tend to break before the fibers break. Such tissue basesheets can be characterized by a relatively low stiffness and have a SEM of about 4 or less.

In another aspect, the invention resides in a soft wet-pressed tissue sheet embossed according to the above mentioned method, which sheet has a Bulk of about 6 cubic centimeters per gram or greater, a Specific Elastic Modulus of about 4 kilometers or less and a Strength of about 500 grams or greater. Alternatively, the invention resides in a soft wet-pressed tissue sheet having a Bulk of about 7 cubic centimeters per gram or greater, a Specific Elastic Modulus of about 3 kilometers or less and a Strength of about 500 grams or greater.

In another aspect, the invention resides in a two-ply tissue product comprising two wet-pressed tissue sheets embossed according to the above mentioned method, which product has a Bulk of about 9 cubic centimeters per gram or greater, a Specific Elastic Modulus of about 2 kilometers or less and a Strength of about 500 grams or greater.

In another aspect, the invention resides in a soft throughdried tissue sheet embossed according to the above mentioned method, which sheet has a Bulk of about 9 cubic centimeters per gram or greater, a Specific Elastic Modulus of about 3 kilometers or less, such as about 2 kilometers or less, and a Strength of about 500 grams or greater.

Suitable tissue basesheets for purposes herein include paper sheets useful for products such as facial tissue, bath tissue, paper towels, dinner napkins, and the like. These sheets can be layered or blended (nonlayered), although the greatest economic benefit can be obtained using blended sheets having a high short fiber content because a product approaching layered quality can be made from a blended basesheet. However, layered sheets can also be improved as well. The tissue basesheets preferably have at least about 20 dry weight percent short fibers, more preferably at least about 40 dry weight percent short fibers, and still more preferably at least about 60 dry weight percent short fibers. Short fibers are natural or synthetic papermaking fibers having an average length of about 2 millimeters (0.08 inch) or less. Generally, short fibers include hardwood fibers such as eucalyptus, maple, birch, aspen and the like. Long fibers are natural or synthetic papermaking fibers having an average length of about 2.5 millimeters (0.1 inch) or greater. Such long fibers include softwood fibers such as pine, spruce and the like.

The basis weight of the tissue sheets of this invention can be from about 5 to about 100 grams per square meter,

more specifically from about 10 to about 70 grams per square meter, and still more specifically from about 20 to about 50 grams per square meter.

The tissue sheets of this invention may also be characterized in part by a machine-direction stretch of less than about 30 percent, more specifically from about 10 to about 25 percent, and still more specifically from about 15 to about 20 percent.

The pair of embossing rolls useful herein can be made of steel or rubber. The male embossing roll of the pair contains discrete "male" embossing elements which protrude from the surface of the embossing roll. The female embossing roll of the pair has corresponding "female voids", sometimes referred to as female "elements", which are recessed from the surface of the embossing roll and are positioned and sized to intermesh with the male elements of the other roll. In operation, the intermeshing embossing elements do not perforate the basesheet.

The nip between the embossing rolls can be operated with a fixed gap, fixed load, press pulse, constant nip width, or other such common operating conditions well known in the embossing art. It will herein be referred to as a fixed gap, meaning that the elements do not bottom out as they are engaged. The fixed gap spacing between the embossing rolls will be affected by the relative size and shape of the male elements and the female voids, as well as the basis weight or thickness of the sheet(s) being embossed.

In general, at least 15 discrete, intermeshing male elements per square centimeter (100 per square inch) is preferred to adequately emboss the surface, more specifically from about 30 to about 95 elements per square centimeter (from about 200 to about 600 per square inch), and still more specifically from about 45 to about 75 per square centimeter (from about 300 to about 500 per square inch). While round or generally oval-shaped elements are preferred for surface fiber feel quality, the cross-sectional shape of the male elements can be any shape, provided that the elements are distinct, which means that the elements are not ridges or lines but are instead individual protrusions surrounded by land area on the embossing roll. The shape of the female voids generally corresponds to that of the male elements, but need not be the same. The size of the female void must be sufficiently large to accept the male element and the tissue sheet.

The width and length of the male elements are preferably less than or equal to the average fiber length of the short fiber species within the sheet. Specifically, the width and length of the male elements can be less than about 2.5 millimeters, more specifically from about 0.25 to about 2 millimeters, and still more specifically from about 0.75 to about 1.25 millimeter. As used herein, the width and length of the embossing elements are sometimes collectively referred to as the "size" of the elements as viewed in cross-section. The width and length can be the same or different.

25

45

The distance between the male elements on the surface of the roll also is preferably less than or equal to the average short fiber length. Specifically, the distance between the male elements is less than about 2.5 millimeters, more specifically from about 0.25 to about 2.0 millimeters, and still more specifically from about 0.75 to about 1.25 millimeters.

As previously mentioned, the female embossing roll has a pattern of depressions or voids adapted to accommodate the intermeshing male elements. When the male elements are aligned with the female voids prior to engagement, the distance between the sidewalls of the male elements and the sidewall of the female voids at zero engagement is referred to, as mentioned above, as the "accommodation". The terminology pertaining to the embossing method of this invention is further described in connection with Figure 7. The degree of accommodation can be from about 0.075 to about 1.25 millimeter, more specifically from about 0.25 to about 0.75 millimeter. In general, accommodation has a significant impact on the Strength loss of the embossing process. As the accommodation decreases, the tissue sheet is subjected to greater shear forces and hence a greater chance of losing Strength.

As mentioned above, the "embossing level", also referred to as the "roll engagement", is the distance the male element penetrates the corresponding female void This distance will in large part determines the Bulk gain imparted by the embossing process. The embossing level can be from about 0.1 to about 1 millimeter, more specifically from about 0.25 to about 0.5 millimeter.

The male elements and female voids are unmatched, which means that they are not mirror images of each other. The unmatched elements can differ in size, depth, and/or sidewall angles. Sidewall angles are preferably in the range of from about 15° to about 25° and are preferably substantially the same for the male elements and the corresponding female voids. In such a case, it is also preferred that the size of the top of the male element be larger than the size of the bottom of the female void to prevent the male element from contacting the bottom of the female void. Unmatched embossing elements include elements produced by laser-engraving rubber rolls. Unmatched elements provide greater flexibility in terms of embossing level and accommodation. The use of laser-engraved embossing rolls is described in greater detail in US-5 356 364 filed April 17, 1992 in the names of J. S. Veith et al. entitled "Method For Embossing Webs".

In designing the size of the male embossing elements and female voids, it is preferable that the length and width of the male elements is equal to or greater than the distance between surrounding adjacent male elements. If the element size is maintained constant, the density of the elements (the number of elements per square centimeter) can be increased by decreasing the space between the elements. Alternatively, if the density of the elements is maintained constant, the element size can be increased by decreasing the space between the elements. A tissue sheet embossed

in accordance with this invention can approach a one-sided feel (both sides of the embossed sheet feel substantially the same) if the accommodation, element size, female roll land distance and the number of elements per unit length are properly balanced (see Figure 7 for a clarification of these parameters). More specifically, the following equation represents a linear inch (25.4 millimeters) of the embossing pattern taken in cross-section:

$$(2A + B + C) \times D = 25.4$$
 millimeters (1 inch)

where:

5

10

15

- A = accommoda:tion (required on both sides of the element), expressed in millimeters;
- B = element size, length or width, expressed in millimeters;
- C = female roll land distance, expressed in millimeters; and
- D = number of elements per lineal 25.4 millimeters (1 inch).

Some of the parameters have minimum requirements. For example, the land distance of the female roll is limited to a minimum of 0.1016 millimeter (0.004 inch) due to embossing roll manufacturing limitations and for maintaining adequate integrity to run the embossing process. It is also not desirable to design embossing patterns with less than 0.0762 millimeter (0.003 inch) accommodation, which would limit the embossing level and thereby limit bulk generation.

A key to eliminating or minimizing two-sidedness is providing an embossing pattern in which the length and width of the male elements is greater than or equal to the distance between male elements. Stated in terms of the parameters defined above:

$$B \ge (2A + C)$$

Any combination of accommodation and female roll land distance can be used as long as the above formula is met.

By way of example, set forth below are several combinations of embossing element design parameters within the scope of this invention and which are suitable for producing a one-sided sheet (all dimensions in millimeters):

•	5	
9	0	

40

45

50

55

30

Elements per 25,4 Mil- limeters	Accomodation	Element Size	Female Roll Land Dis- tance
10	0.0762	2.286	0.1016
10	0.5842	1.270	0.1016
10	0.0762	1.270	1.1176
25	0,0762	0.762	0.1016
25	0.2032	0.508	0.1016
25	0.0762	0.508	0.3556

In the drawings:

Figure 1 is a cross-sectional view of a brushed sample mounted between glass slides, illustrating the protruding fiber ends which are exposed as the test sample is folded over the glass cover slip.

Figure 2 is a plan view of a prior art butterfly embossing pattern, illustrating the shape of the male embossing elements.

Figure 3 is a plan view of an embossing pattern useful in accordance with this invention (magnified 2X), illustrating the shape and spacing of the male embossing elements.

Figure 4 is a plan view of an embossing pattern not useful in accordance with this invention (magnified 2X), illustrating the shape and spacing of the male embossing elements.

Figure 5 is a plan view of another embossing pattern useful in accordance with this invention (magnified 2X), illustrating the shape and spacing of the male embossing elements.

Figure 6 is a plan view of another embossing pattern useful in accordance with this invention (magnified 2X), illustrating the shape and spacing of the male embossing elements.

Figure 7 is a schematic view of a tissue sheet being embossed in accordance with this invention, illustrating the

intermeshing of the male embossing elements and corresponding female voids.

5

10

Figure 8 is a plot of Bulk versus SEM for commercially available single-ply tissue products (wet-pressed and throughdried), illustrating how the method of this invention can impart throughdried-like qualities to a wet-pressed sh et. (This plot includes the data from Table 3.)

Figure 9 is a plot similar to that of Figure 8, but illustrating the improvement in Bulk as a function of different embossing levels. (This plot includes the data from Table 4.)

Figure 10 is a plot similar to that of Figure 8, but showing the improvement in Bulk for a different basesheet. (This plot includes the data from Table 5.)

Figure 11 is a plot similar to that of Figure 8, but showing the improvement in Bulk for a throughdried basesheet. (This plot includes the data from Table 8.)

Figure 1 illustrates an end view of a test sample showing fiber ends protruding from the surface of the sample. Shown is the test sample 3, a cover slip 11 over which the test sample is folded, and two glass slides 12 and 13 which protect the sample and firmly hold it in place for viewing. Also schematically depicted are numerous fiber ends 14 protruding from the surface of the test sample at the point where the sample is folded over the edge of the cover slip.

Figure 2 is a plan view of a prior art decorative butterfly embossing pattern produced on laser-engraved embossing rolls, illustrating the shape of the male embossing elements. The male butterfly embossing elements had a line thickness of 0.71 millimeter (0.028 inch), a depth of 1.6 millimeter (0.062 inch) and a sidewall angle of 22°. The matching female void was 1.4 millimeter wide (0.057 inch), 1.3 millimeter deep (0.053 inch) and had a 19° sidewall angle. The butterfly was 17.5 millimeters long (0.6875 inch) by 15.9 millimeters wide (0.625 inch), and there were 0.2131 butterflies per square centimeter (1.375 butterflies per square inch). Seven different elements made up the butterfly pattern to provide an embossing area of about 10 percent.

Figure 3 is a plan view of an embossing pattern useful in accordance with this invention, illustrating the size and spacing of the male embossing elements. For this pattern, the male elements had a height (or depth) of 0.76 millimeter, a length of 1.52 millimeter and a width of 0.508 millimeter, hence having a length:width ratio of 3:1. The major axes of the elements were oriented at an angle of 65° relative to the circumferential direction of the roll. There were an average of 0.5 elements per millimeter in the axial direction of the roll and an average of 1.1 element per millimeter in the circumferential direction of the roll, resulting in an element density of 57 discrete elements per square centimeter. The female roll in the nip contained corresponding voids positioned to receive the male elements having a depth of 0.81 millimeter, a length of 2.03 millimeters and a width of 1.02 millimeter. The voids were correspondingly oriented with the major axes at an angle of 65° to the circumferential direction of the roll. The land area between the voids was 0.15 millimeter with an accommodation between the intermeshing elements of 0.25 millimeter. The side wall angle of the male element and the female void was 18°. The embossing area was about 45 percent.

Figure 4 is a plan view of an embossing pattern not useful in accordance with this invention, illustrating the shape and spacing of the male embossing elements. For this pattern, the male elements had a depth of 8.6 millimeters (0.34 inch), an element surface area of 0.035 square centimeter (0.0055 square inch), a sidewall angle of 33°, an element density of 8.5 elements per square centimeter (55 elements per square inch), and a repeat unit length of 7.6 millimeters (0.3 inch). The embossing area was about 30 percent. Figure 5 is a plan view of another embossing pattern useful in accordance with this invention, illustrating the size and spacing of the male embossing elements. For this pattern, there were 39.6 discrete intermeshing elements per square centimeter (256 elements per square inch). Each element was 0.84 millimeter long (0.033 inch) by 0.84 millimeter wide (0.033 inch) and had an 18° sidewall angle. The corresponding female void was 1.09 millimeter long (0.043 inch) by 1.09 millimeter wide (0.043 inch), leaving 0.127 millimeter (0.005 inch) accommodation between the two intermeshing elements. The land distance between the female voids was 0.20 millimeter (0.008 inch) for a total of 0.46 millimeter (0.018 inch) between the individual male elements. The embossing area was about 28 percent.

Figure 6 is a plan view of another embossing pattern useful in accordance with this invention (magnified 2X), illustrating the shape and spacing of the male embossing elements. The male roll had approximately 50.2 discrete protruding male embossing elements per square centimeter (324 per square inch). Each element was 0.38 millimeter wide (0.015 inch) by 0.76 millimeter long (0.030 inch), with every other element rotated 90°. The sidewall angle of the elements was 20°. The distance between the male protruding elements was 1.01 millimeter (0.040 inch). The corresponding female void was 1.14 millimeter wide (0.045 inch) by 1.52 millimeter long (0.060 inch), matching the orientation of the male element. The accommodation between the intermeshing elements was 0.38 millimeter (0.015 inch) and the land distance between the female voids was 0.25 millimeter (0.010 inch). The embossing area was about 15 percent.

Figure 7 is a schematic view of a tissue sheet being embossed in accordance with this invention, illustrating the intermeshing relationship of the male elements and female voids. Shown is the female embossing roll 21, the male embossing roll 22 and the tissue basesheet 23 being embossed. The male embossing element 24 is shown as partially engaging the female void 25. The degree of roll engagement or embossing level is indicated by the distance 26, which is the distance that the male element penetrates the female void. The depth of the male element is indicated by refer-

ence numeral 27. The depth of the female void is indicated by reference numeral 28. The size of the male element (length or width, depending on the orientation of the element relative to the cross-sectional view) is indicated by reference numeral 30. The size of the female void is similarly indicated by reference numeral 31. The size of the bottom or base of the female void is indicated by reference numeral 32. The land area between the female voids is indicated by reference numeral 34. The sidewall angle of the male elements and female voids is measured relative to a line which is perpendicular to the surface of the rolls. The sidewall angle of the male element is shown as reference numeral 33. The accommodation is the distance between the male element sidewalls and the female void sidewalls at zero engagement. Although the elements in Figure 7 are not at zero engagement, the accommodation would be the distance between points 35 and 36 at zero engagement. As the elements are engaged, the distance between the sidewalls decreases, causing shearing of the tissue to create a permanent deformation and a corresponding bulk increase. It is believed to be important that the male elements do not inelastically compress the tissue between the top 37 of the male element and the bottom 38 of the female void. That is to say, referring to Figure 7, that the distance 39 is not less than the thickness of the tissue.

Figure 8 is a plot of Bulk versus SEM for commercially available single-ply tissue products, illustrating how the method of this invention can be used to impart throughdried-like qualities to a wet-pressed sheet. The commercially available wet-pressed tissues are labelled "W". The commercially available throughdried tissues are labelled "T". Note that the throughdried products have a lower SEM than the wet-pressed tissues, indicating greater softness. In general, the throughdried tissues also have greater Bulk. The point labelled M₀ is a wet-pressed control sample, and the point labelled M₁ is the product resulting from applying the method of this invention to the control sample. (See Table 3 for specific data). Note that the Bulk of the wet-pressed product has been elevated to the level of the throughdried products.

Figure 9 is a plot containing the same commercially available wet-pressed and throughdried products of Figure 8, but illustrating the improvements in Bulk for differing levels of embossing roll engagement (embossing level). Specifically, the wet-pressed tissue control sample is represented as " M_o " was subjected to the method of this invention at different levels of engagement. The resulting products are represented by points M_2 , M_3 and M_4 . Specific data is presented in Table 4. As shown, these products possess a combination of softness, Strength and Bulk not exhibited by the prior art wet-pressed products.

Figure 10 is a plot similar to Figure 8, illustrating the improvement in Bulk attained by applying the method of this invention to a different control wet-pressed basesheet. As before, the starting material is designated M_0 and the product of this invention is designated as M_5 . Specific data is presented in Table 5.

Figure 11 is a plot similar to Figure 8, illustrating the improvement in Bulk attained by applying the method of this invention to a throughdried control basesheet using different embossing levels.

The control basesheet is designated as X_0 and the resulting products are designated X_1 , X_2 , and X_3 . As shown, the throughdried products can be elevated to Bulk levels not exhibited by the commercially available throughdried products. Specific data is presented in Table 8.

Examples

35

To further illustrate the invention, the methods of making the tissue products of this invention plotted in Figures 8, 9, 10 and 11 will be described in detail below.

Example 1. A blended tissue sheet was made with 70% Caima sulfite eucalyptus and 30% northern softwood kraft and was embossed between unmatched laser-engraved rubber embossing rolls having an embossing pattern as illustrated in Figure 3 having an embossing level of 0.20 millimeter (0.008 inch). The embossed sheets were plied together with a like sheet by crimping the edges of the sheets to produce a two-ply product having a finished basis weight of 44 grams per square meter (gsm), a Bulk of 7.04 cubic centimeters per gram and a Strength of 784 grams per 7.62 centimeters. After embossing in accordance with this invention, the resulting tissue had improved softness.

Example 2. A one-ply, blended, wet-pressed tissue basesheet was made with a furnish comprising 70% Cenibra eucalyptus bleached kraft and 30% northern softwood kraft having a dryer basis weight of 27.5 grams per square meter (16.2 pounds per 2880 square feet) and a finished basis weight of 33.9 grams per square meter (19.9 pounds per 2880 square feet). The machine speed was 396 meters per minute (1300 feet per minute), using no refiner or wet strength agents. The resulting basesheet had a machine direction stretch of 24 percent, a Bulk of 4.2 cubic centimeters per gram, a Strength of 1025 grams and a SEM of 2.30 kilometers. This basesheet is designated as the Control sample.

The Control basesheet was embossed with a matched steel embossing pattern as illustrated in Figure 4. The base-sheet was embossed at incremental levels to generate a Bulk gain/Strength loss relationship. Table 1 below shows the resulting data. (For all of the data listed in the following tables, "Embossing Level" is expressed in millimeters, "Basis Weight" is expressed in grams per square meter, "Strength" is expressed in grams per 76.2 millimeters of sample width, "Bulk" is expressed in cubic centimeters per gram, "SEM" (Specific Elastic Modulus) is expressed in kilometers, and "RATIO" is the ratio of the percent increase in Bulk divided by the percent decrease in Strength.

TABLE 1

SAMPLE **EMBOSSING LEVEL BASIS WEIGHT** STRENGTH BULK SEM **RATIO** Control 33.89 1025 4.20 2.30 1 0.1778 31.85 1022 4.15 3.08 0 2 0.2794 30.57 962 4.32 3.75 0.47 3 0.3810 31.31 847 4.70 2.64 0.69 4 0.4826 30.57 689 4.90 2.52 0.51

In all cases the resulting basesheet did <u>not</u> meet all three of the criteria of Strength, softness (SEM), and Bulk for a premium tissue product.

The Control basesheet was also embossed with a set of unmatched laser-engraved rolls having a butterfly pattern as shown in Figure 2.

Again, the basesheet was embossed at various levels to obtain a Bulk gain/Strength loss relationship. Table 2 below shows the resulting data:

TABLE 2

SAMPLE **EMBOSSING LEVEL BASIS WEIGHT** STRENGTH **BULK SEM RATIO** Control 33.89 1025 4.20 2.30 1 0.2540 31.33 1025 4.46 2.91 0 2 0.3810 31.75 945 4.56 2.38 1.10 3 0.5080 31.85 832 4.46 3.19 0.33 4 0.6350 32.50 737 5.24 2.00 0.88

Again, the resulting basesheet did <u>not</u> meet all three of the criteria for Strength, softness (SEM) and Bulk for a premium product. Sample 2 did exhibit a Ratio greater than 1, but this was obtained because the Bulk increase was so low (9%) that the Strength was not significantly impacted. Also, the differences in Bulk and Strength values are within basesheet variability and testing deviation.

Example 3. The same Control basesheet described in Example 2 was embossed in accordance with this invention with a laser-engraved micro pattern as illustrated in Figure 3 to obtain the Strength, softness (SEM) and Bulk of a premium tissue product. Table 3 below shows the resulting data:

TABLE 3

SAMPLE	EMBOSSING LEVEL	BASIS WEIGHT	STRENGTH	BULK	SEM	RATIO
M _o		33.89	1025	4.20	2.30	-
M ₁	0.3556	30.02	629	7.36	1.80	1.95

The resulting basesheet met the premium criteria of Strength, softness (SEM) and Bulk.

The micro embossing pattern described above was used to emboss a different control basesheet at various embossing levels. All process conditions were as described in Example 2 except for the furnish blend, in which a portion of the eucalyptus was substituted with Caima eucalyptus, which is a sulfite pulp exhibiting less bonding potential than the Cenibra eucalyptus. The overall make-up of the blended base sheet was 35 percent Cenibra eucalyptus/35 percent Caima eucalyptus/30 percent northern softwood kraft. The resulting data is listed in Table 4 below:

50

5

10

20

25

30

40

TABLE 4

SAMPLE	EMBOSSING LEVEL	BASIS WEIGHT	STRENGTH	BULK	SEM	RATIO
Mo		32.40	1092	4.23	2.67	-
M ₂	0.2540	30.24	815	6.80	2.02	2.39
M ₃	0.2794	29.16	765	7.14	2.16	2.30
M ₄	0.3048	30.02	731	7.36	2.00	2.24

Again, the resulting basesheet met the premium criteria of Strength, softness (SEM) and Bulk.

The same micro embossing pattern described above was applied to a Control basesheet made as described in Example 2, but having a lower dryer basis weight of 24.7 grams per square meter (14.6 pounds per 2880 square feet). The overall make-up of the blended Control basesheet was 70 percent Cenibra eucalyptus and 30 percent northern softwood kraft. The embossing level was 0.25 millimeter (0.010 inch). The resulting data is listed in Table 5 below:

TABLE 5

SAMPLE	EMBOSSING LEVEL	BASIS WEIGHT	STRENGTH	BULK	SEM	RATIO
M ₀		29.92	935	4.41	2.16	-
M ₅	0.2540	28.41	666	6.52	1.92	1.66

The result was that the embossed basesheet met the premium criteria of Strength, softness (SEM) and Bulk.

Example 4.

5

10

20

25

30

40

45

55

A different wet-pressed Control basesheet was embossed in accordance with this invention between a pair of laserengraved embossing rolls having the embossing pattern described and illustrated in connection with Figure 8. The Control basesheet was produced on a crescent former and was layered. The wire side (dryer side) layer was 100 percent
Cenibra eucalyptus and the roll side (air side) layer was a blend of 40 percent northern softwood kraft and 60 percent
broke. The weight ratio of the two layers was 50/50. The dryer basis weight of the Control basesheet was 12.1 grams
per square meter (7.17 pounds per 2880 square feet). The basesheet was embossed with the dryer side of the basesheet being contacted by the male embossing roll and a roll engagement of 0.25 millimeter (0.010 inch). Like embossed
basesheets were then plied together, dryer side out, by crimping the edges together to form a two-ply tissue. The resulting data is listed in Table 6 below:

TABLE 6

SAMPLE	EMBOSSING LEVEL	BASIS WEIGHT	STRENGTH	BULK	SEM	RATIO
Control		30.23	743	8.35	1.90	-
1	0.2540	27.96	550	9.01	1.73	0.30

Both the Control and embossed samples met the premium criteria of Strength, softness (SEM) and Bulk, but the embossed sample had improved softness and Bulk, although there was a decrease in Strength.

Example 5. A one-ply, throughdried, layered basesheet was produced using a twin-wire former. This Control base-sheet was embossed between a laser-engraved male embossing roll (having the butterfly embossing pattern described in Figure 2) and a 60 durometer smooth rubber roll over a range of loads to obtain a Strength loss/Bulk gain relationship. The resulting data is listed in Table 7 below:

TABLE 7

SAMPLE	EMBOSSING LEVEL	BASIS WEIGHT	STRENGTH	BULK	SEM	RATIO
Control		28.77	996	6.89	2.58	-
1	23.8125	28.77	779	7.77	2.06	0.52
2	25.4000	28.41	739	7.78	2.23	0.50
3	30.1625	28.57	572	8.45	2.58	0.53

The Control sheet met the Strength, softness (SEM) and Bulk criteria for a premium tissue product. Embossing the basesheet with the butterfly pattern resulted in a 42% Strength loss for a 23% Bulk increase with no change in SEM. The percent Bulk increase per percent Strength decrease was 0.55.

For comparison, the one-ply throughdried basesheet listed above was embossed in accordance with this invention using a set of intermeshing laser-engraved rolls having the embossing pattern described in Figure 6. The basesheet was embossed over a range of roll engagements to produce a Strength loss/Bulk increase relationship. The resulting data is listed in Table 8 below:

TABLE 8

SAMPLE	EMBOSSING LEVEL	BASIS WEIGHT	STRENGTH	BULK	SEM	RATIO
X _o		28.77	996	6.89	2.58	-
X ₁	0.2032	28.14	852	7.58	2.00	0.70
X ₂	0.3048	27.79	725	9.41	1.81	1.34
X ₄	0.4064	27.63	555	11.03	1.66	1.36

Micro embossing the same sheet in accordance with this invention resulted in a 60% increase in Bulk for the same 44% decrease in Strength as the butterfly with a 36% decrease in SEM.

35 Claims

5

10

20

25

30

40

- 1. A method of making a soft tissue sheet comprising passing a tissue sheet (23) through a nip formed between male (22) and female (21) embossing rolls having about 15 or more discrete, intermeshing, unmatched, embossing elements (24,25) per square centimeter of surface which deflect the sheet (23) perpendicular to its plane, wherein the embossing elements cause, when they are engaged, shearing of the tissue to create a permanent deformation without perforation of said tissue, so that the percent Bulk increase divided by the percent Strength decrease is about 1 or greater.
- 2. The method of Claim 1 wherein the number of discrete intermeshing elements (24,25) is from about 30 to about 95 per square centimeter.
 - The method of Claim 2 wherein the number of discrete intermeshing elements (24,25) is from about 45 to about 75 per square centimeter.
- 4. The method of any of Claims 1 to 3 wherein the percent Bulk increase divided by the percent Strength decrease is from about 1 to about 4.
 - The method of Claim 4 wherein the percent Bulk increase divided by the percent Strength decrease is from about 2 to about 3.
 - 6. The method of any of Claims 2 to 5 wherein said intermeshing embossing elements (24,25) are engaged at an embossing level (26) of from about 0.1 to about 1 millimeter.

- 7. The method of Claim 6 wherein the intermeshing embossing elements (24,25) are engaged at an embossing level of from about 0.25 to about 0.5 millimeter.
- 8. The method of any of the preceding claims wherein the embossing elements have a degree of accommodation of from about 0.075 to about 1.25 millimeter.
 - The method of Claim 8 wherein the embossing elements have a degree of accommodation of from about 0.25 to about 0.75 millimeter.
- 10. The method of any of the preceding claims wherein the unmatched elements have substantially equal sidewall angles.
 - 11. The method of Claim 10 wherein the sidewall angles are from about 15° to about 25°.
- 15 12. The method of Claim 11 wherein the top of the male element is larger than the bottom of the female element.
 - 13. The method of any of Claims 2 to 12 wherein the embossing pattern further satisfies the formula:

$$B \ge (2A + C)$$
,

20

25

wherein

"A" is the accommodation,

"B" is the element width, and

"C" is the female roll land distance between female voids

wherein A, B and C are determined along the same cross-sectional direction.

- 14. A soft wet-pressed tissue sheet embossed by the method according to any of Claims 1 to 13, wherein said sheet has a Bulk of about 6 cubic centimeters per gram or greater, a Specific Elastic Modulus of about 4 kilometers or less, and a Strength of about 500 grams or greater.
 - 15. The tissue sheet of Claim 14 having a Bulk of about 7 cubic centimeters per gram or greater and a Specific Elastic Modulus of about 3 kilometers or less.

35

- 16. The tissue sheet of Claim 14 having a Bulk of about 7 cubic centimeters per gram or greater and a Specific Elastic Modulus of about 2 kilometers or less.
- 17. A two-ply tissue product comprising two wet-pressed tissue sheets embossed by the method according to any of
 Claims 1 to 13, wherein said product has a Bulk of about 9 cubic centimeters per gram or greater, a Specific Elastic
 Modulus of about 2 kilometers or less and a Strength of about 500 grams or greater.
 - 18. A soft throughdried tissue sheet embossed by the method according to any of Claims 1 to 13, wherein said sheet has a Bulk of about 9 cubic centimeters per gram or greater, a Specific Elastic Modulus of about 3 kilometers or less and a Strength of about 500 grams or greater.
 - 19. The tissue sheet of Claim 18 having a Specific Elastic Modulus of about 2 kilometers or less.

Patentansprüche

50

55

45

1. Verfahren zur Herstellung eines weichen Tissue-Bogens, bei dem ein Tissue-Bogen (23) durch einen Walzenspalt geführt wird, der zwischen einer Patrizenprägewalze (22) und einer Matrizenprägewalze (21) gebildet ist, die etwa 15 oder mehr einzelne, ineinandergreifende, ungleiche Prägeelemente (24,25) pro cm² Fläche aufweisen, welche die Bögen (23) senkrecht zu dessen Ebene wölben, wobei die Prägeelemente, wenn diese sich in Eingriff befinden, ein Scheren des Tissues bewirken, um eine permanente Deformation ohne Perforation des Tissues zu erzeugen, so daß der prozentual Bauschanstieg geteilt durch die prozentuale Festigkeitsabnahme einen Wert von etwa 1 oder mehr ergibt.

- Verfahren gemaß Anspruch 1, bei dem die Anzahl an einzelnen ineinandergreifenden Elementen (24,25) etwa 30 bis etwa 95 pro cm² beträgt.
- Verfahren gemaß Anspruch 2, bei dem die Anzahl der einzelnen ineinandergreifenden El mente (24,25) etwa 45
 bis etwa 75 pro cm² beträgt.
 - 4. Verfahren gemäß einem der Ansprüche 1 bis 3, bei dem der prozentuale Bauschanstieg geteilt durch die prozentuale Festigkeitsabnahme einen Wert von etwa 1 bis etwa 4 ergibt.
- Verfahren gemäß Anspruch 4, bei dem der prozentuale Bauschanstieg geteilt durch die prozentuale Festigkeitsabnahme einen Wert von etwa 2 bis etwa 3 ergibt.
 - 6. Verfahren gemäß einem der Ansprüche 2 bis 5, bei dem die ineinandergreifenden Prägeelemente (24,25) mit einem Prägeniveau (26) von etwa 0,1 bis etwa 1 mm ineinandergreifen.

7. Verfahren gemäß Anspruch 6, bei dem die ineinandergreifenden Prägeelemente (24,25) mit einem Prägeniveau von etwa 0,25 bis etwa 0,5 mm ineinandergreifen.

- 8. Verfahren gemäß einem der vorhergehenden Ansprüche, bei dem die Prägeelemente einen Akkommodationsgrad von etwa 0,075 bis etwa 1,25 mm aufweisen.
 - Verfahren gemäß Anspruch 8, bei dem die Prägeelemente einen Akkommodationsgrad von etwa 0,25 bis etwa 0,75 mm aufweisen.
- 10. Verfahren gemäß einem der vorhergehenden Ansprüche, bei dem die ungleichen Elemente im wesentlichen gleiche Seitenwandwinkel aufweisen.
 - 11. Verfahren gemäß Anspruch 10, bei dem die Seitenwandwinkel etwa 15° bis etwa 25° betragen.
- 30 12. Verfahren gemäß Anspruch 11, bei dem das Oberteil des Patrizenelements größer ist als der Boden des Matrizenelements.
 - 13. Verfahren gemäß einem der Ansprüche 2 bis 12, bei dem das Prägemuster des weiteren folgende Gleichung erfüllt:

 $B \ge (2A + C),$

wobei

15

35

40

45

55

"A" die Akkommodation,

"B" die Elementbreite und

"C" der Patrizenwalzenstegabstand zwischen Patrizenhohlräumen darstellen,

wobei A, B und C entlang derselben Querschnittsrichtung bestimmt werden.

14. Weicher naßgeprägter Tissue-Bogen, welcher entsprechend des Verfahrens gemäß einem der Ansprüche 1 bis 13 geprägt ist, wobei der Bogen eine Bauschigkeit von etwa 6 cm³/g oder mehr, ein spezifisches elastisches Modul von etwa 4 km oder weniger und eine Festigkeit von etwa 500 g oder größer aufweist.

- 15. Tissue-Bogen gemäß Anspruch 14, mit einer Bauschigkeit von etwa 7 cm³/g oder größer und einem spezifischen elastischen Modul von etwa 3 km oder weniger.
 - 16. Tissue-Bogen gemäß Anspruch 14 mit einer Bauschigkeit von etwa 7 cm³/g oder größer und einem spezifischen elastischen Modul von etwa 2 km oder weniger.
 - 17. Zweilagiges Tissue-Produkt mit zwei naßgepreßten Tissue-Bögen, welcher mittels des Verfahrens gemaß einem der Ansprüche 1 bis 13 geprägt ist, wobei das Produkt eine Bauschigkeit von etwa 9 cm³/g oder mehr, ein spezifisches Modul von etwa 2 km oder weniger und eine Festigkeit von etwa 500 g oder mehr aufweist.

- 18. Weicher mittels eines Durchtrittstrocknungsverfahrens getrockneter Tissue-Bogen, welcher mittels des Verfahrens gemaß einem der Ansprüche 1 bis 13 geprägt ist, wobei die Bogen eine Bauschigkeit von etwa 9 cm³/g oder mehr, ein spezifisches elastisches Modul von etwa 3 km oder weniger und eine Festigkeit von etwa 500 g oder mehr aufweist.
- 19. Tissue-Bogen gemäß Anspruch 18 mit einem spezifischen elastischen Modul von etwa 2 km oder weniger.

Revendications

20

- 10. Procédé de fabrication d'une feuille de papier absorbant mince et doux qui consiste à faire passer une feuille de papier absorbant mince (23) au travers d'un interstice formé entre des rouleaux de gaufrage mâles (22) et femelles (21) ayant environ 15 éléments de gaufrage s'interpénétrant (24,25) non appariés et séparés, ou davantage, par cm² de surface, qui incurvent la feuille (23) perpendiculairement à son plan, dans lequel les éléments de gaufrage provoquent, lorsqu'ils se pénètrent, un cisaillement de la feuille pour créer une déformation permanente sans perforation de ladite feuille, de telle sorte que le pourcentage d'augmentation du Bouffant divisé par le pourcentage de réduction de la Résistance est d'environ 1 ou davantage.
 - Procédé selon la revendication 1, dans lequel le nombre d'éléments séparés s'interpénétrant (24,25) est compris entre environ 30 et environ 95 par cm².
 - Procédé selon la revendication 2, dans lequel le nombre d'éléments séparés s'interpénétrant (24,25) est compris entre environ 45 et environ 75 par cm².
- 4. Procédé selon l'une des revendications 1 à 3, dans lequel le pourcentage d'augmentation du Bouffant divisé par le pourcentage de réduction de la Résistance est compris entre environ 1 et environ 4.
 - Procédé selon la revendication 4, dans lequel le pourcentage d'augmentation du Bouffant divisé par le pourcentage de réduction de la Résistance est compris entre environ 2 et environ 3.
- 6. Procédé selon l'une quelconque des revendications 2 à 5, dans lequel lesdits éléments de gaufrage s'interpénétrant (24,25) s'interpénètrent à un niveau de gaufrage (26) compris entre environ 0,1 et environ 1 mm.
 - 7. Procédé selon la revendication 6, dans lequel lesdits éléments de gaufrage s'interpénétrant (24,25) s'interpénètrent à un niveau de gaufrage (26) compris entre environ 0,25 et environ 0,5 mm.
 - Procédé selon l'une quelconque des revendications précédentes, dans lequel les éléments de gaufrage ont un degré de correspondance compris entre environ 0,075 et environ 1,25 mm.
- Procédé selon la revendication 8, dans lequel les éléments de gaufrage ont un degré de correspondance compris
 entre environ 0,25 et environ 0,75 mm.
 - 10. Procédé selon l'une quelconque des revendications précédentes, dans lequel les éléments non appariés ont des angles de parois latérales sensiblement égaux.
- 45 11. Procédé selon la revendication 10, dans lequel les angles des parois latérales sont compris entre environ 15° et environ 25°.
 - 12. Procédé selon la revendication 11, dans lequel le sommet de l'élément mâle est plus grand que le fond de l'élément femelle.
 - 13. Procédé selon l'une quelconque des revendications 2 à 12, dans lequel le motif de gaufrage satisfait en outre à la formule :

$$B \ge (2A + C)$$
,

55 Où

50

"A" est la correspondance.

"B" est la largeur de l'élément, et

5

15

20

30

35

40

45

50

- "C" est la distance de zone plate du rouleau femelle entre les cavités femelles,
- A, B et C étant déterminées le long de la même direction transversale.
- 14. Feuille de papier absorbant mince, souple et pressé à l'état humide, gaufrée par le procédé selon l'une quelconque des revendications 1 à 13, ladite feuille ayant un Bouffant d'environ 6 cm³ par gramme ou davantage, un Module d'Elasticité Spécifique d'environ 4 km ou moins, et une Résistance d'environ 500 g ou plus.
- 10 15. Feuille de papier absorbant mince selon la revendication 14, ayant un Bouffant d'environ 7 cm³ par gramme ou davantage et un Module d'Elasticité Spécifique d'environ 3 km ou moins.
 - 16. Feuille de papier absorbant mince selon la revendication 14, ayant un Bouffant d'environ 7 cm³ par gramme ou davantage et un Module d'Elasticité Spécifique d'environ 2 km ou moins.
 - 17. Produit à deux jets formé de deux feuilles de papier absorbant mince comprimé à l'état humide, gaufrées par le procédé selon l'une quelconque des revendications 1 à 13, ledit produit ayant un Bouffant d'environ 9 cm³ par gramme ou davantage, un Module d'Elasticité Spécifique d'environ 2 km ou moins et une Résistance d'environ 500 g ou plus.
 - 18. Feuille de tissu absorbant mince et doux séché par soufflage transversal, gaufrée par le procédé selon l'une quelconque des revendications 1 à 13, ladite feuille ayant un Bouffant d'environ 9 cm³ par gramme ou davantage, un Module d'Elasticité Spécifique d'environ 3 km ou moins et une Résistance d'environ 500 g ou plus.
- 19. Feuille de papier absorbant mince selon la revendication 18, ayant un Module d'Elasticité Spécifique d'environ 2 km ou moins.

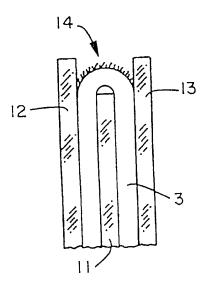
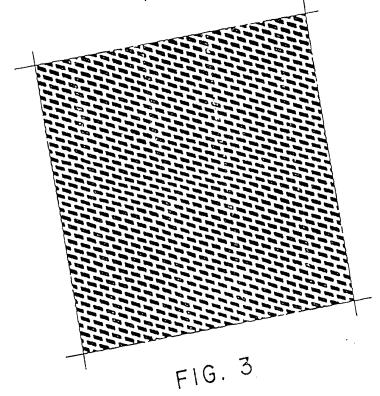
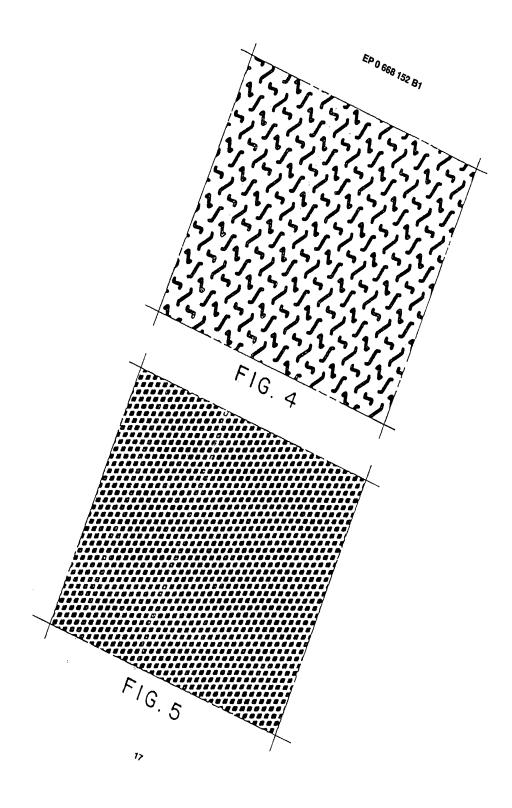


FIG. I





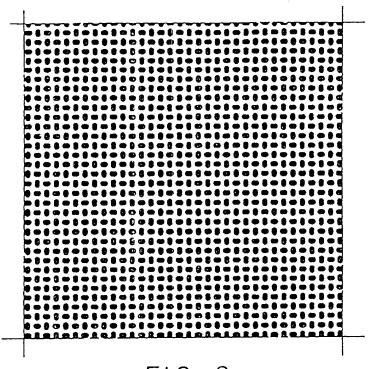
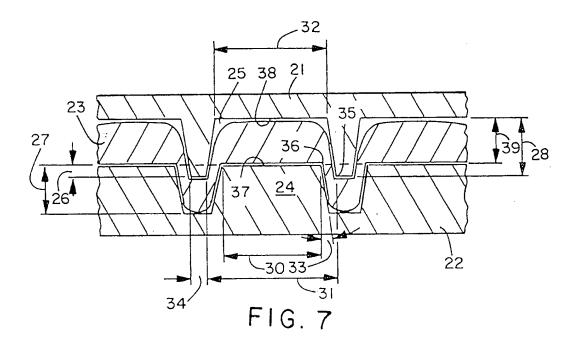
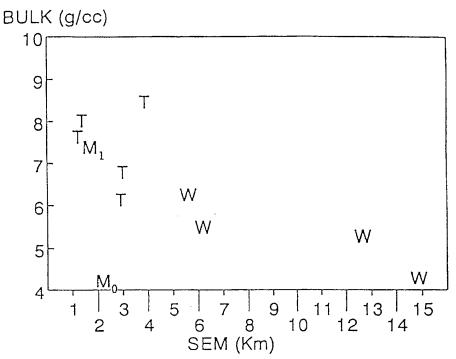


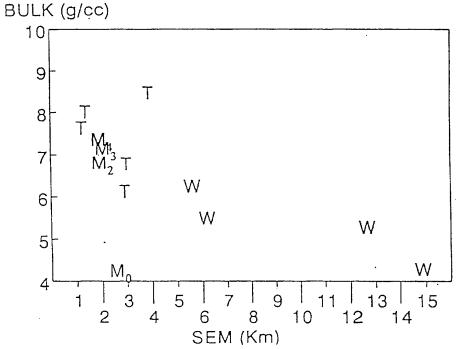
FIG. 6





W=Wet Press T=Through Dried M=Invention

FIG. 8



W=Wet Press T=Through Dried M=Invention

FIG. 9

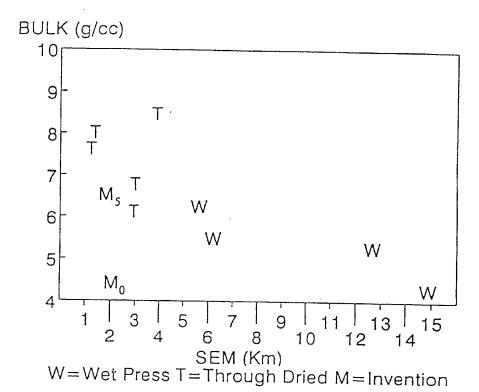


FIG. 10

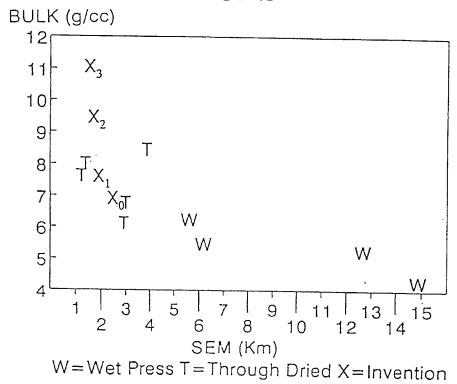


FIG. 11